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## Automated Structure Determination

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From reflection file to fully assigned and validated structure - from dream to reality. We will present new software which needs nothing more than the reflection file, the unit cell dimensions and some information about the crystal symmetry. From this starting point, a variety of tools are employed to solve the structure and then correctly assign the atom types, including hydrogen positions. In most routine cases, a correctly assigned and finished structure, complete with IUCr cif-check report and ready-to-submit ".cif" files will result without the need for intervention. The crystallographic core of this software is provided by G. M. Sheldrick. XS, XD and XL have earned the trust of many crystallographers during decades of wide-spread use in the community. These new tools build on this proven crystallographic basis and are designed to work with the Bruker axS "Apex" software suite. We will present in detail their effectiveness, limitations and detailed roles in the process of fully automatic structure solution and refinement. Issues arising from twinning and disorder as well as incomplete or missing formula and their impact on the system will be discussed.

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## How can we cope with negative scattering density?

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Charge flipping (CF) is an amazingly simple ab initio structure solution method [1,2] that has already attracted useful applications [3-6]. It is based on the existence of extended zero plateaus in the ideal electron density, but not directly on atomicity. The CF algorithm is iterative and works in dual spaces. While in reciprocal space there is a choice of several algorithm variants, the real-space modification always changes the sign of electron density below a threshold  $\delta$ . Although positivity was thought to be a crucial precondition for the CF algorithm, recently we have investigated how well negative scattering density can be tolerated. This negative part can be either physical or artificial and the two cases should be treated separately.

First, let us consider artificial negative density, that occurs due to resolution cutoff or using E's of point-like atoms. Recall that higher resolution and/or lower temperature data simultaneously increase the volume samples of large positive density and suppress negative density, so these parameters will only do good for the efficiency of the algorithm. The effect of sharpening seems more controversial. Our numerical tests show that using point-like atoms further improves the efficiency, the speedup of convergence can be a factor of 10. Considering that most direct methods operate with E's instead of F's, this observation is not surprising. Sharpening is done for its positive effect, in the act of increasing the large positive electron density it actually decreases the effective dimensionality of the problem. Unfortunately, at the same time it also amplifies the artificial negative part of the electron density distribution. At each charge-flipping cycle large negative samples must change their sign that is unnecessary and can be harmful. The two effects of using E's are always coupled. Obviously, using x-ray diffraction data at normal resolutions, the positive effect dominates, and the only harm is the smaller drop of the R-factor at convergence. Now let us turn to the case of physical negative density, for which the main example is the contribution of hydrogen to neutron diffraction. This effect does not disappear with higher resolution data, and should be handled if our aim is ab initio structure solution using neutron data alone. The existence of large zero plateaus of the ideal density is the basis of the CF algorithm, while positivity is forced by flipping all negative density. In the neutron case we have two options for modifying the original algorithm: (i) flip only the  $\pm\delta$  region around zero developing both positive and negative densities, or (ii) run the original version up to the convergence and allow negative densities only afterwards. In our tests both versions worked. This means that the existence of plateaus is so important that CF can work even without positivity. However, version (ii) was more efficient than version (i). This means that positivity can even help the solution of non-positive structures. The likely reason is that opening up negative scattering space too early can mix different  $\pm$ Babinet-solutions, a diffraction data ambiguity of real scattering objects. This research was supported by OTKA T043494.

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